

[NAME OF DOCUMENT] PATENT APPLICATION

[REFERENCE NO.] IDS1608A

[SUBMISSION DATE] April 5, 2004

[ADDRESSED TO] Commissioner of Japanese Patent Office

5 [INTERNATIONAL PATENT CLASSIFICATION] G03F 7/039

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[DEPOSIT ACCOUNT NUMBER] 013619

[DEPOSIT AMOUNT] 16,000 yen

[LIST OF SUBMITTED MATTERS]

25 [NAME OF MATTER] Claims 1 set

[NAME OF MATTER] Specification 1 set

[NAME OF MATTER] Drawing 1 set

2004-111459

[NAME OF MATTER] Abstract 1 set

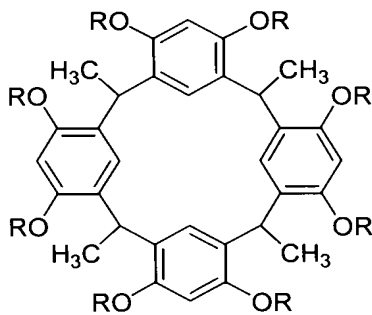
[GENERAL POWER OF ATTORNEY NUMBER] 0200132

[Name of Document] CLAIMS

[Claim 1]

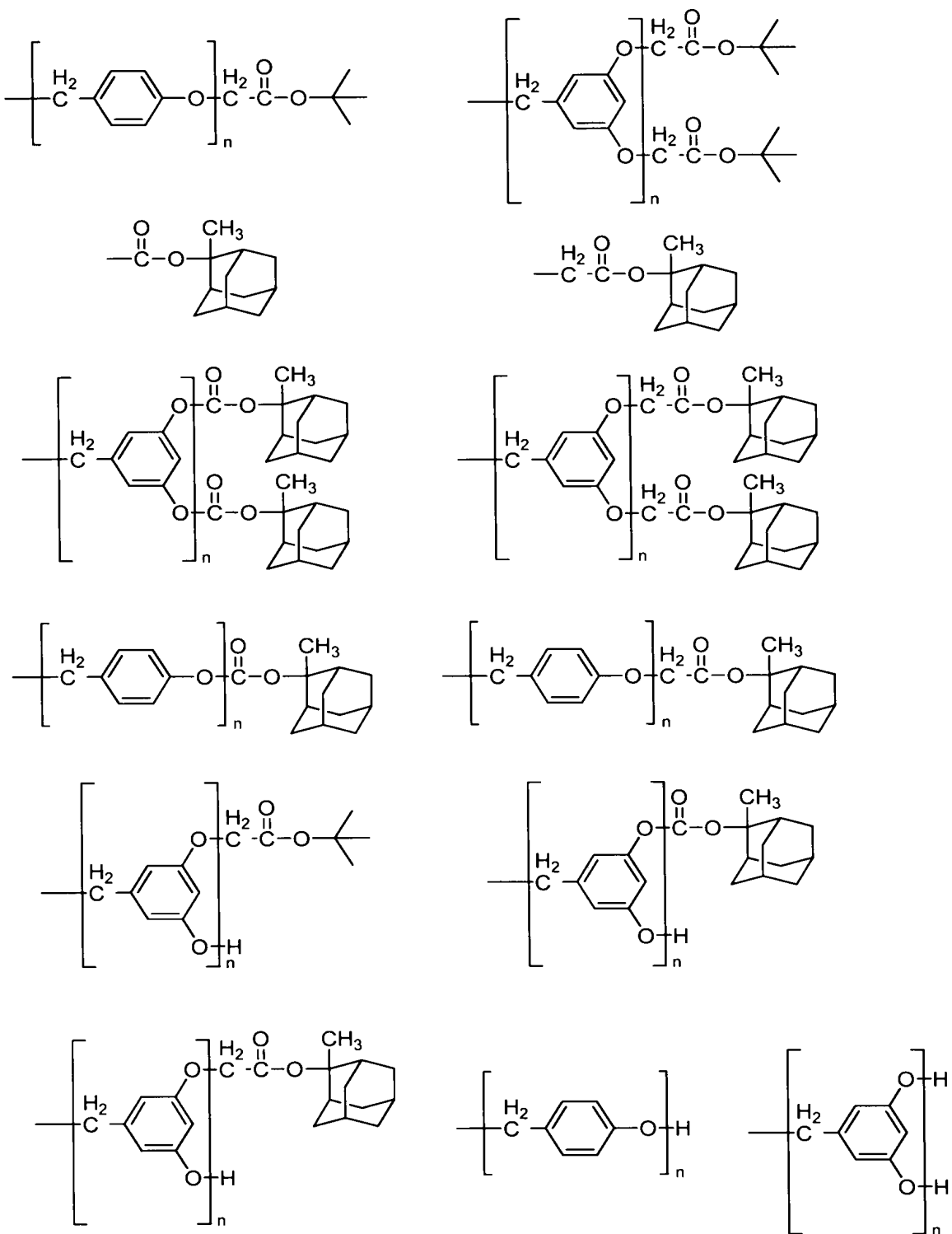
A calixresorcinarene compound shown by the following formula (1),

5



(1)

wherein R individually represents a hydrogen atom, a  
 1-tetrahydropyranyl group, a 1-tetrahydrofuryl group, or one  
 10 or more organic groups selected from the group consisting of  
 the organic groups shown by the following formulas,



wherein n individually represents an integer of 1 to 50,

provided that a compound in which R is selected only from a hydrogen atom, a 1-tetrahydropyranyl group, and a 1-tetrahydrofuranyl group is excluded.

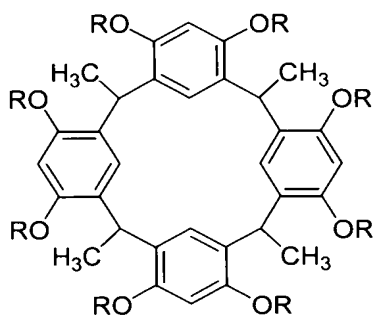
5 [Claim 2]

A purification method of a calixresorcinarene compound comprising washing the compound according to claim 1 with an acidic aqueous solution and processing the washed compound with an ion-exchange resin.

10

[Claim 3]

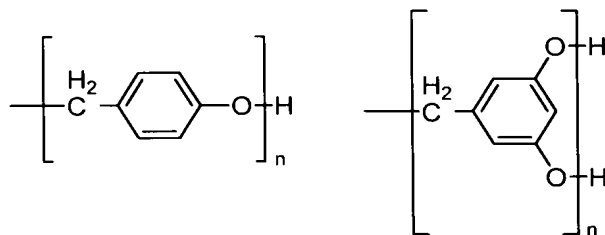
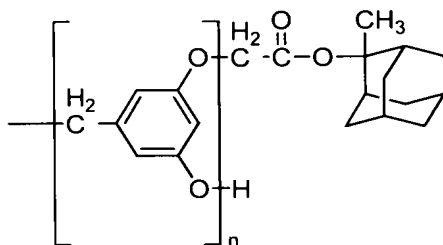
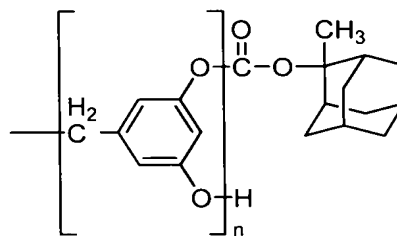
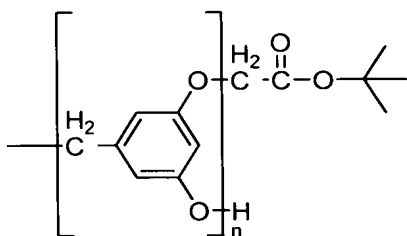
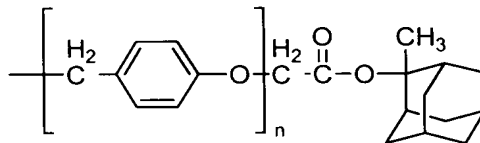
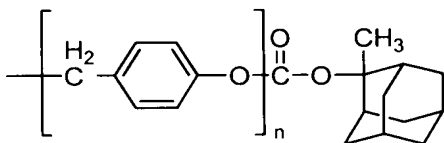
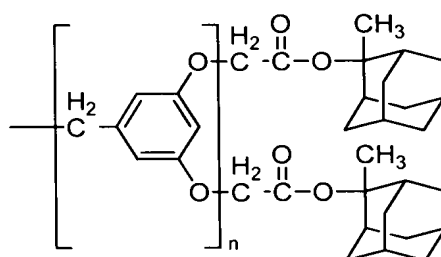
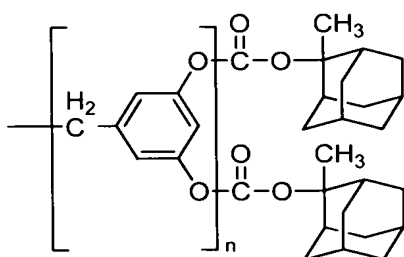
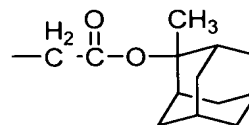
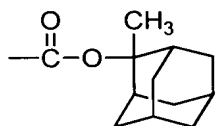
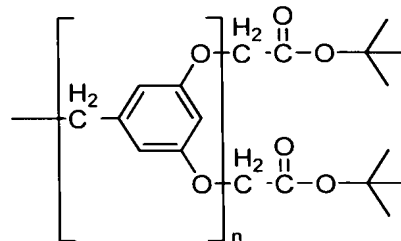
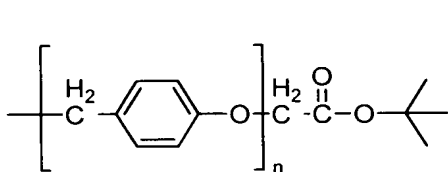
A photoresist base material for extreme ultraviolet radiation and/or an electron beam comprising the calixresorcinarene compound shown by the following formula (1).



(1)

15

wherein R individually represents a hydrogen atom, a 1-tetrahydropyranyl group, a 1-tetrahydrofuranyl group, or one or more organic groups selected from the group consisting of  
20 the organic groups shown by the following formulas,



wherein n individually represents an integer of 1 to 50,

provided that a compound in which R is selected only from a hydrogen atom, a 1-tetrahydropyranyl group, and a 1-tetrahydrofuranlyl group is excluded.

5 [Claim 4]

A photoresist composition for extreme ultraviolet radiation and/or an electron beam comprising the photoresist base material according to claim 3 and a solvent.

10 [Claim 5]

The photoresist composition according to claim 4, further comprising a photoacid generator.

[Claim 6]

15 The photoresist composition according to claim 4 or 5, further comprising a basic organic compound as a quenching agent.

[Claim 7]

20 A purification method of a photoresist base material comprising washing the photoresist base material according to claim 3 with an acidic aqueous solution and processing the washed photoresist base material with an ion-exchange resin.

25 [Claim 8]

A microfabrication method by lithography using the photoresist composition according to any one of claims 4 to 6.

[Claim 9]

A semiconductor device prepared using the photoresist composition according to any one of claims 4 to 6.



[Name of Document] SPECIFICATION

[Title of the Invention] CALIXRESORCINARENE COMPOUNDS,  
PHOTORESIST BASE MATERIALS FOR EXTREME ULTRAVIOLET RADIATION  
AND/OR AN ELECTRON BEAM COMPRISING THE COMPOUNDS , AND  
5 COMPOSITIONS THEREOF

[Technical Field]

[0001]

The invention relates to a novel calixresorcinarene  
compound, a photoresist substrate for extreme ultraviolet  
10 radiation and/or an electron beam comprising the compound used  
in the electricity and the electronic field such as a  
semiconductor, the optical field, and the like, and a  
composition of the same.

[Background Art]

15 [0002]

Lithography using extreme ultraviolet radiation (EUV) or  
an electron beam is useful as a high productivity and high  
resolution microfabrication method in the manufacture of  
semiconductors and the like. Development of a high sensitivity  
20 and high resolution photoresist used in lithography is desired.  
Improvement of sensitivity is essential for a photoresist used  
in the lithographic technique from the viewpoint of  
productivity of desired detailed patterns, high resolution, and  
the like.

25 [0003]

As a photoresist used for microfabrication using extreme  
ultraviolet radiation, a chemically-amplified

polyhydroxystyrene-based photoresist used for microfabrication using a known KrF laser, for example, can be given. This resist is known to be usable for microfabrication to a degree of about 50 nm. However, if patterns more detail  
5 than 50 nm, which is the greatest merit of the microfabrication using extreme ultraviolet radiation, are produced using this resist, problems such as low sensitivity, large line edge roughness, and a large amount of resist out-gas occur. The resist thus cannot necessarily sufficiently derive excellent  
10 performance inherent to extreme ultraviolet radiation. Therefore, development of a photoresist exhibiting higher performance has been demanded.

[0004]

In order to respond to such a demand, a method of using  
15 a chemically amplified positive-tone photoresist with a higher photoacid generator concentration as compared with other resist compounds has been proposed (e.g., refer to Patent Document 1). This Patent Document discloses a photoresist comprising a hydroxystyrene/styrene/t-butyl acrylate terpolymer base  
20 material, a photoacid generator of which at least about 5 wt% of the total solid content is di(t-butylphenyl)iodonium ortho-trifluoromethylsulfonate, lactate of tetrabutylammonium hydroxide, and ethyl lactate in examples. However, no specific results such as a line width obtained by  
25 using extreme ultraviolet radiation are described. Therefore, the results attainable by using this composition have been thought that microfabrication to the extent of 100 nm, shown

in an example in which electron beams were used, is a limit in terms of line edge roughness. An overreaction of the basic material due to addition of an excessive amount of photoacid generator, specifically, over-diffusion of acids to non-exposed areas may be a cause of this limitation.

[Patent Document 1] JP-A-2002-055457

[Disclosure of the Invention].

[Subject to be solved by the Invention]

[0005]

The invention has been achieved in view of this situation and has an object of providing a novel calixresorcinarene compound, a photoresist base material comprising the compound enabling ultra microfabrication using extreme ultraviolet radiation and/or an electron beam, while exhibiting high sensitivity, high resolution, and low line edge roughness, and a composition of the same.

[0006]

The inventors have conducted extensive studies in order to achieve the above object and found that a problem that occurs in microfabrication using a conventional photoresist is a decrease in sensitivity of the photoresist, which is caused by a molecular shape of a high molecular compound conventionally used as the photoresist base material, reactivity due to the structure of protective groups in the molecular structure of the photoresist base material, and basic impurities in residues originating from a reaction agent or catalyst used during preparation of the photoresist base material or basic

impurities mixed in the photoresist base material from human bodies or environment.

In particular, a photoacid generator is sometimes used at a high concentration in a photoresist when the absorbance of extreme ultraviolet radiation and an electron beam passing  
5 through a photoresist layer is high and the strength of a light source is low. If a small amount of a basic impurity mixes in such a photoresist, such an impurity neutralizes protons generated from the acid generator and a desired reaction does  
10 not proceed. This problem is particularly remarkable in a calixresorcinarene-based photoresist.

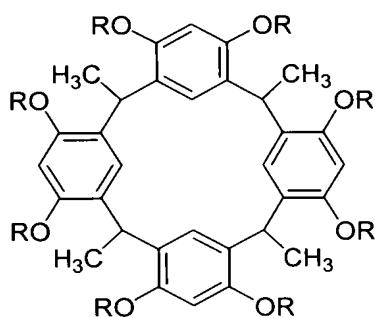
The inventors have found a photoresist base material free from these problems and exhibiting high sensitivity, high resolution, and low line edge roughness, leading to completion  
15 of the invention.

[Means for solving the Subject]

[0007]

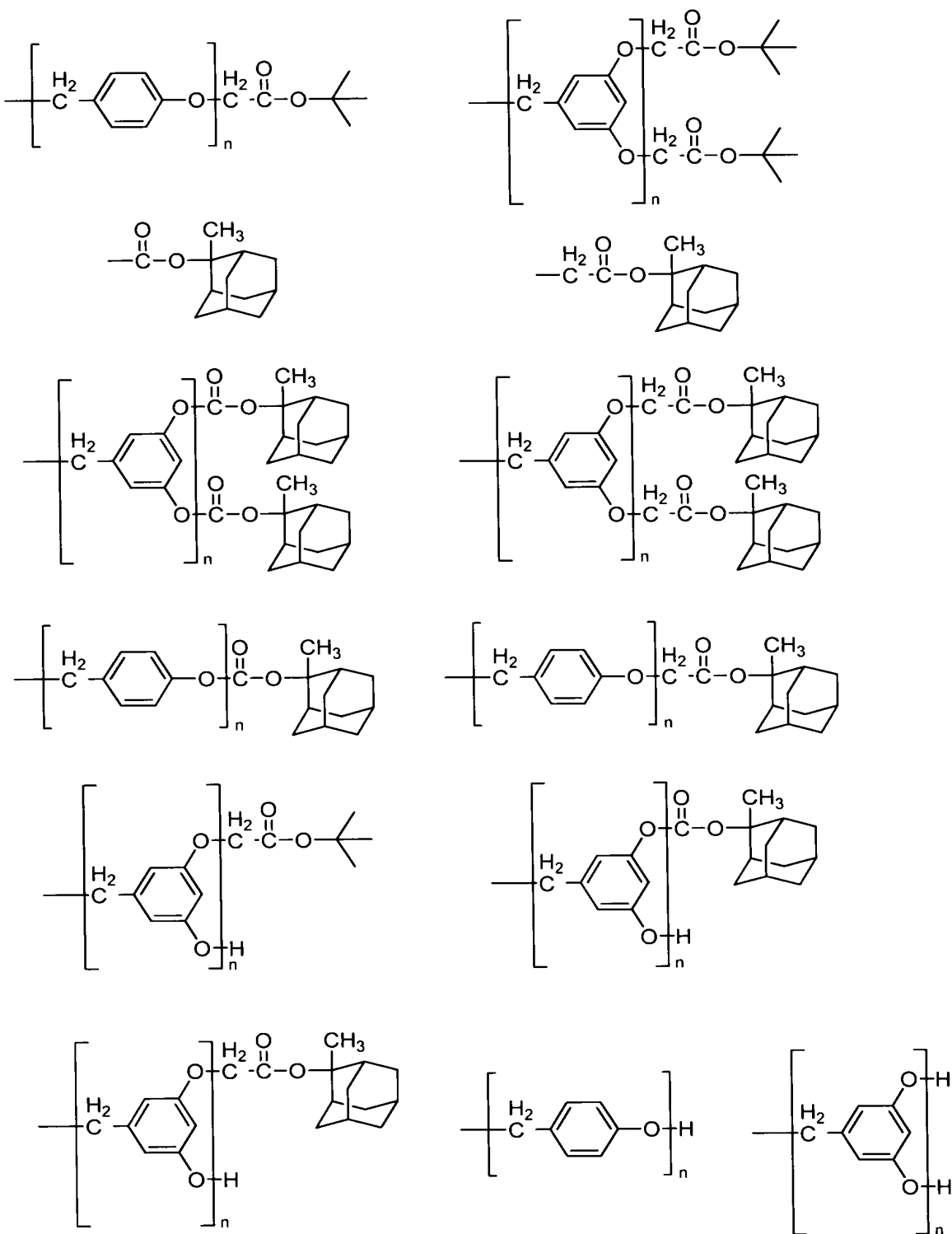
According to the invention, the following calixresorcinarene compounds and the like are provided.

- 20 1. A calixresorcinarene compound shown by the following formula (1),



(1)

wherein R individually represents a hydrogen atom, a  
 1-tetrahydropyranyl group, a 1-tetrahydrofuranyl group, or one  
 5 or more organic groups selected from the group consisting of  
 the organic groups shown by the following formulas,



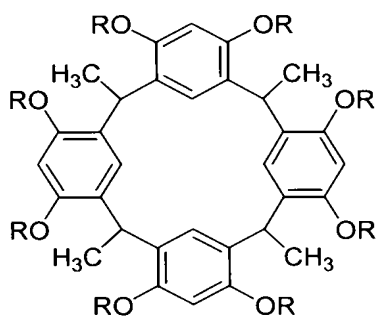
wherein  $n$  individually represents an integer of 1 to 50,

provided that a compound in which R is selected only from a hydrogen atom, a 1-tetrahydropyranyl group, and a 1-tetrahydrofuranlyl group is excluded.

2. A purification method of a calixresorcinarene compound comprising washing the compound according to 1 with an acidic aqueous solution and processing the washed compound with an ion-exchange resin.

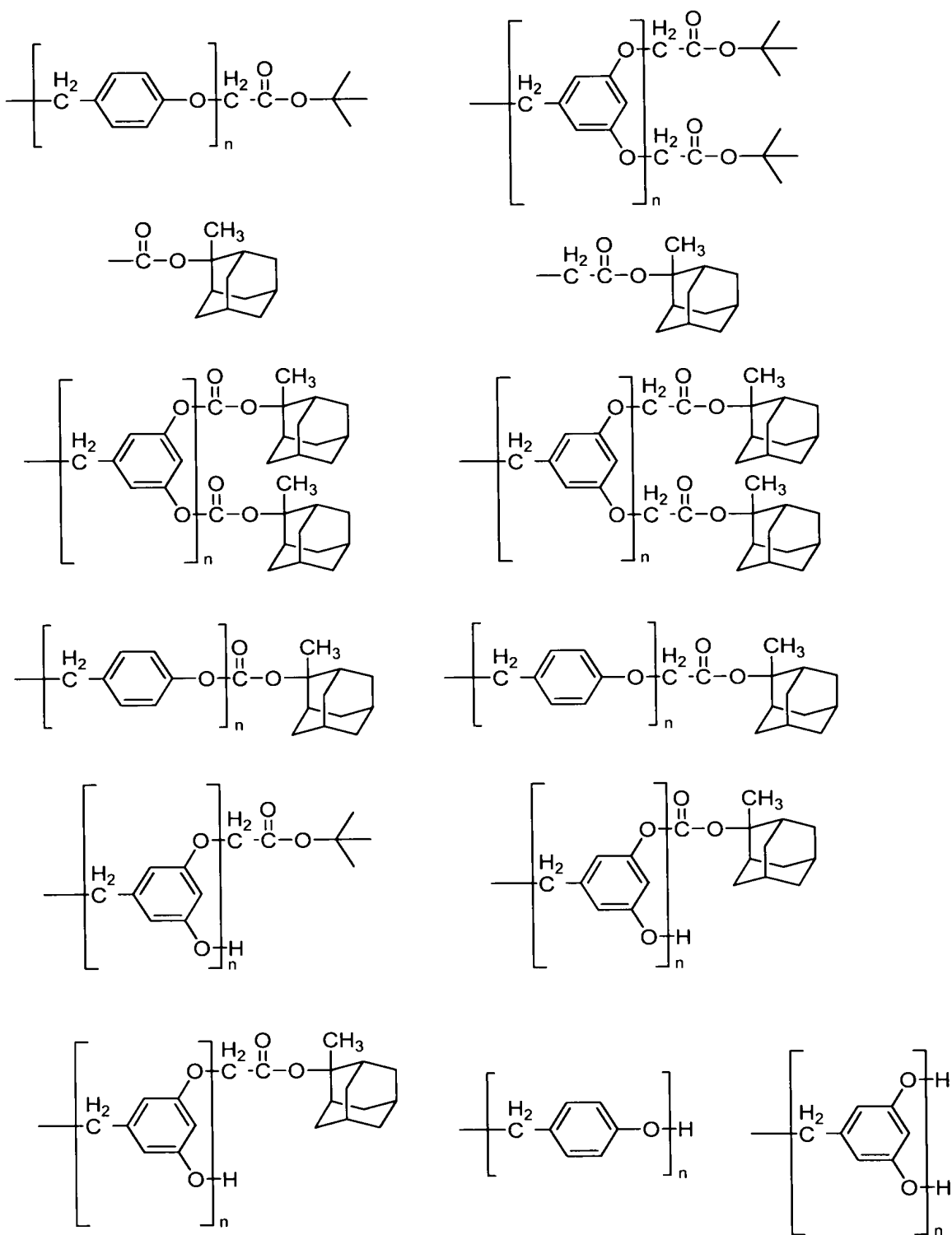
3. A photoresist base material for extreme ultraviolet radiation and/or an electron beam comprising the

10 calixresorcinarene compound shown by the following formula (1).



(1)

wherein R individually represents a hydrogen atom, a 1-tetrahydropyranyl group, a 1-tetrahydrofuranlyl group, or one or more organic groups selected from the group consisting of the organic groups shown by the following formulas,



wherein n individually represents an integer of 1 to 50,



provided that a compound in which R is selected only from a hydrogen atom, a 1-tetrahydropyranyl group, and a 1-tetrahydrofuranly group is excluded.

4. A photoresist composition for extreme ultraviolet radiation and/or an electron beam comprising the photoresist base material according to 3 and a solvent.

5. The photoresist composition according to 4, further comprising a photoacid generator.

6. The photoresist composition according to 4 or 5, further comprising a basic organic compound as a quenching agent.

7. A purification method of a photoresist base material comprising washing the photoresist base material according to 3 with an acidic aqueous solution and processing the washed photoresist base material with an ion-exchange resin.

8. A microfabrication method by lithography using the photoresist composition according to any one of 4 to 6.

9. A semiconductor device prepared using the photoresist composition according to any one of 4 to 6.

[Advantageous Effect of the Invention]

[0008]

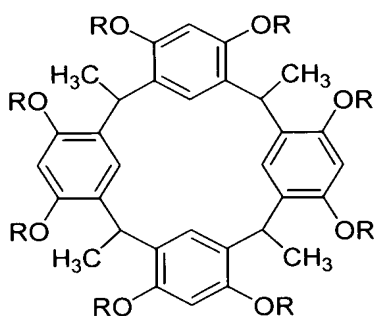
According to the invention, a novel calixresorcinarene compound, a photoresist base material comprising the compound enabling ultra microfabrication using extreme ultraviolet radiation and/or an electron beam, while exhibiting high sensitivity, high resolution, and low line edge roughness, and a composition of the same can be provided.

[Best Mode for Carrying out the Invention]

[0009]

The photoresist base material for extreme ultraviolet radiation and/or an electron beam of the invention will be described in detail.

5        The photoresist base material of the invention comprises a calixresorcinarene compound shown by the following formula (1),

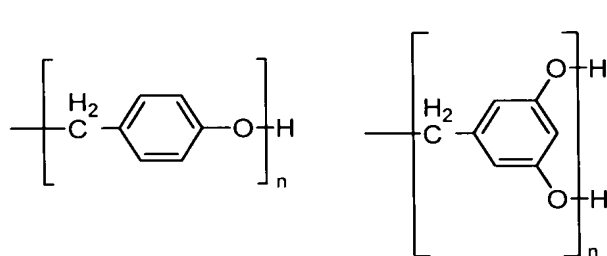
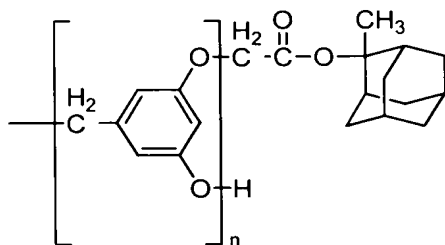
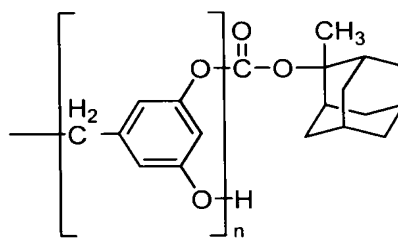
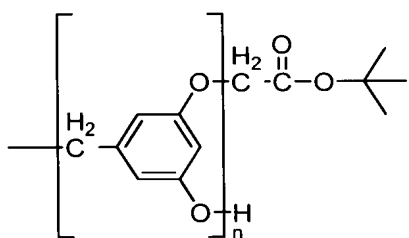
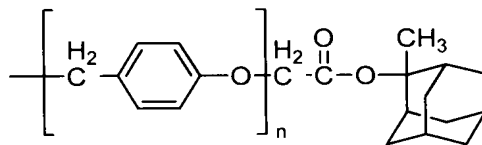
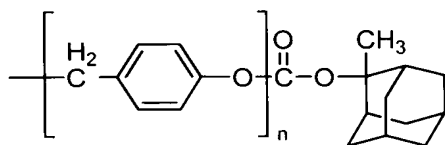
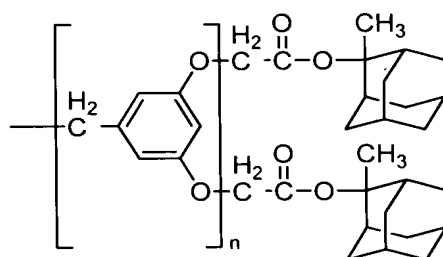
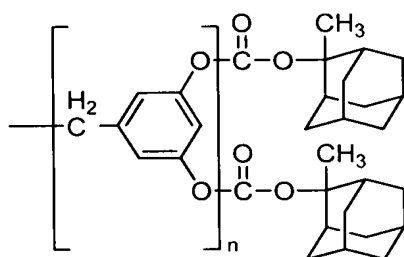
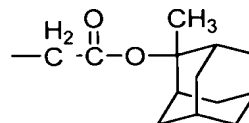
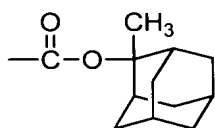
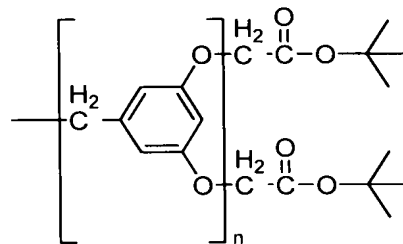
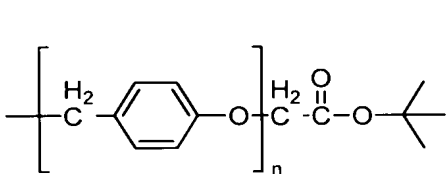


(1)

10

wherein R individually represents a hydrogen atom, a 1-tetrahydropyranyl group, a 1-tetrahydrofuryl group, or one or more organic groups selected from the group consisting of the organic groups shown by the following formulas,

15



wherein n is individually an integer of 1 to 50, provided that

a compound in which R is selected only from a hydrogen atom, a 1-tetrahydropyranyl group, and a 1-tetrahydrofuranyl group is excluded, and provided further that one to seven R groups among eight are preferably hydrogen atoms.

5 [0010]

When the compounds of the invention are used as a base material, the amount of basic impurities, such as ammonia, alkali metal ions (e.g., Li, Na, K, etc.), alkaline earth metal ions (e.g., Ca, Ba, etc.), and the like, contained in these  
10 compounds before purification is preferably reduced to 1/10 or less.

A specific content of basic impurities is preferably 10 ppm or less, and still more preferably 2 ppm or less.

A 10 ppm or less basic impurity content of these compounds  
15 can dramatically increase the sensitivity of the photoresist base material to extreme ultraviolet radiation and an electron beam and, as a result, ensures fabrication of minute patterns by lithographic processing of the photoresist composition.

[0011]

20 The compounds used by the invention are synthesized by combining known reactions. In this instance, the compounds obtained by the reaction can be purified by appropriately separating any impurities contained in the compounds using a known method.

25 [0012]

In the invention, the basic impurity content of these compounds can be reduced to 10 ppm or less by purifying these

compounds by washing with an acidic aqueous solution, followed by processing with an ion-exchange resin. In this instance, an optimal acidic aqueous solution and ion-exchange resin can be appropriately selected according to the amount and type of the basic impurities to be removed, the type of the compound to processed, and the like. In the invention, an acetic acid aqueous solution with a concentration of 0.01 to 10 mol/liter is preferably used as the acidic aqueous solution and a cation-exchange resin is preferably used as the ion-exchange resin. A particularly preferable method of purification comprises washing with an acetic acid aqueous solution as the acidic aqueous solution, followed by processing with a cation-exchange resin.

[0013]

Such a compound is useful as a photoresist base material, particularly, as a photoresist base material used for ultra-microfabrication using extreme ultraviolet radiation or electron beams.

Since the compounds used in the invention are in the state of amorphous under the conditions in which the compounds are used as the photoresist base material, usually at room temperature, the compounds are preferable base materials used in a photoresist composition exhibiting excellent applicability and capability of producing photoresist film with excellent strength.

In addition, the compounds used in the invention usually have an average diameter of the molecule of less than a desired

pattern size, specifically, less than the value of line edge roughness required for the size of 100 nm or less, and particularly 50 nm or less. For this reason, if these compounds are used as a base material, the compound can suppress the line edge roughness to 2 nm or less, and preferably 1 nm or less ( $3\sigma$ ), when used for processing in a range of 20 to 50 nm, featuring the ultra-microfabrication using extreme ultraviolet radiation or an electron beam.

[0014]

These compounds may be used as a photoresist base material either alone or, to the extent that the effect of the invention is not impaired, in combination of two or more. In addition, compounds produced by combining two or more of these compounds by optional substituents may be used either alone or, to the extent that the effect of the invention is not impaired, in combination of two or more.

[0015]

The photoresist base material of the invention can be used as one of the components of the photoresist composition.

The composition of the invention is a liquid composition comprising the photoresist base material comprising the above-described compounds and a solvent. It is required that the composition is liquid in order to uniformly apply a photoresist to a substrate and the like which are to be processed by ultra-microfabrication.

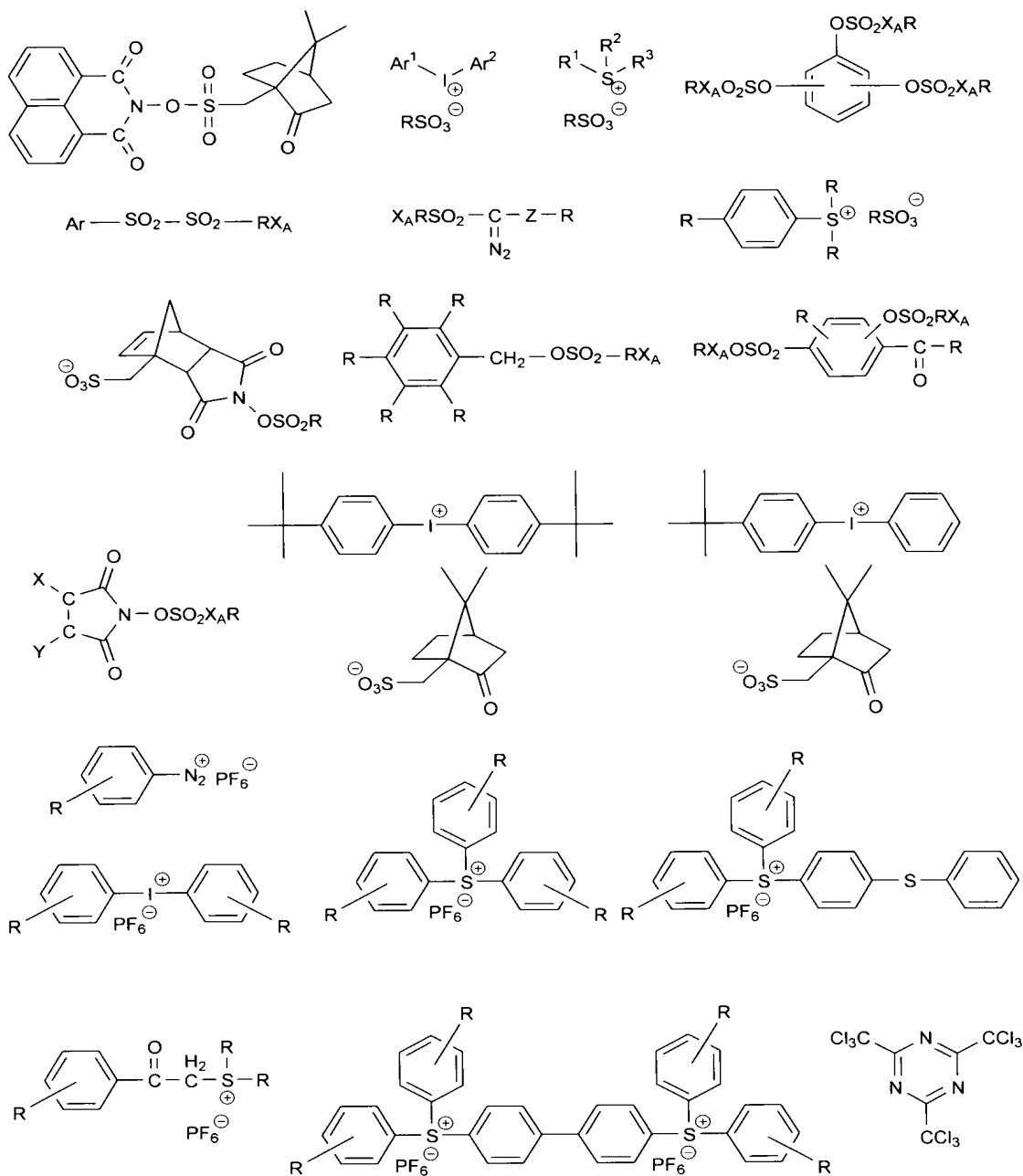
[0016]

Since the molecule of the photoresist base material of the

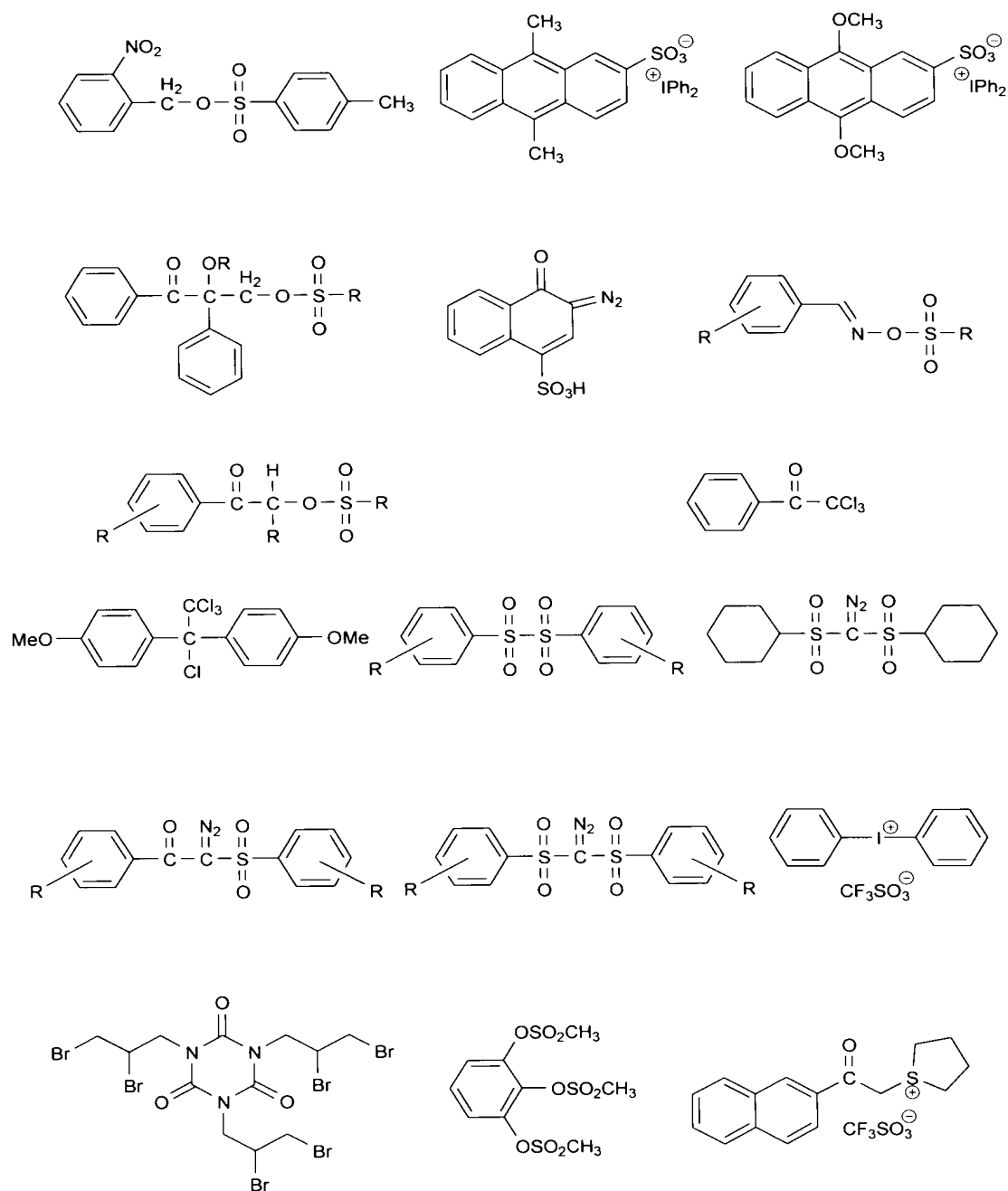
invention contains a chromophore active to radiation such as extreme ultraviolet radiation and/or an electron beam, the photoresist base material can exhibit performance as a photoresist by itself. Therefore, it is unnecessary to add an additive. However, if promotion of the performance as a photoresist is desired, a photoacid generator (PAG) or a photobase generator (PBG) can be added as a chromophore, as required.

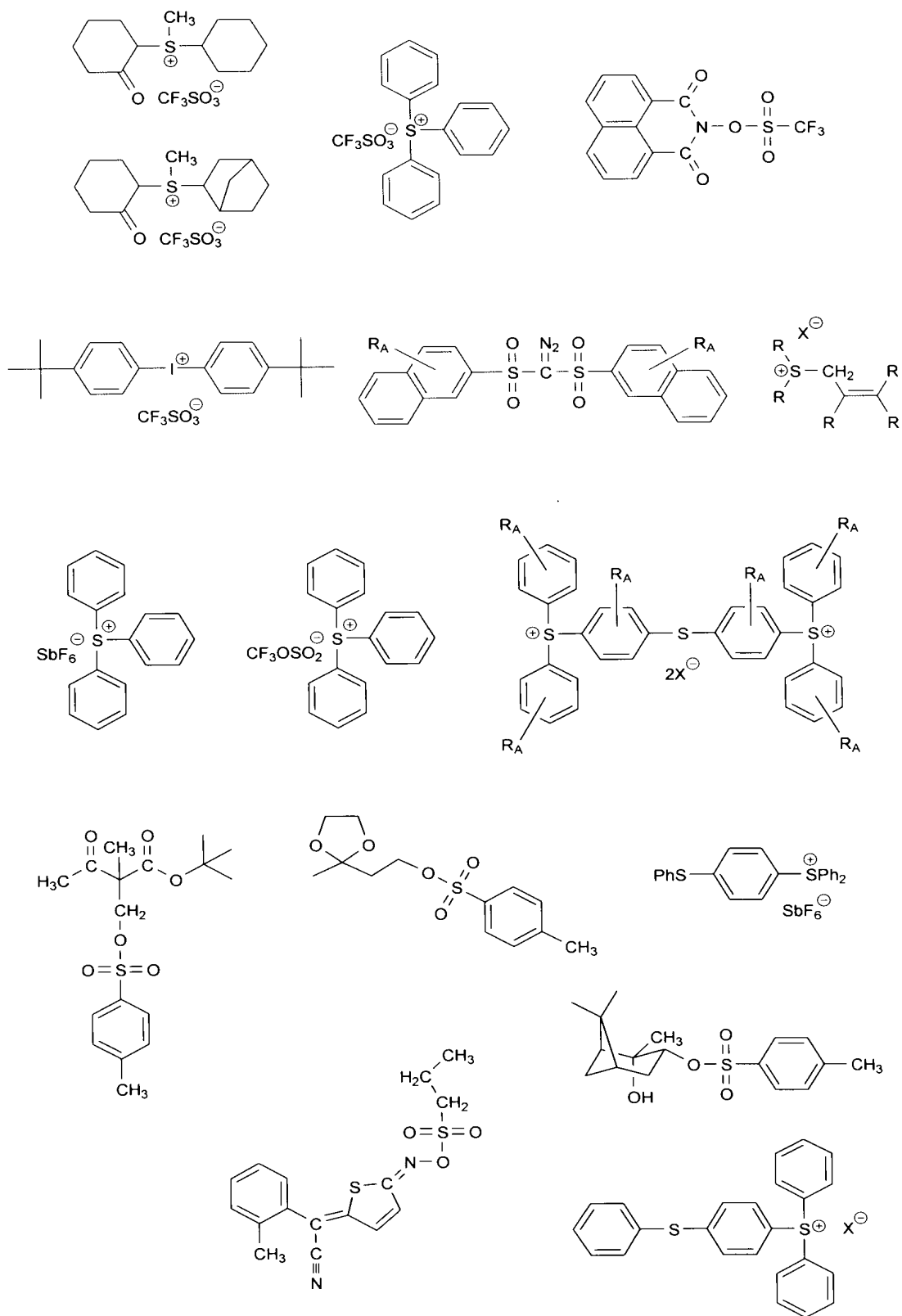
[0017]

As the PAG, in addition to known compounds of which the structures are exemplified below, compounds having the same effect can be commonly used. These compounds can be appropriately selected according to the type of substrate, a desired shape, size, and the like of minute patterns.









In the above formulas, Ar, Ar<sup>1</sup>, and Ar<sup>2</sup> are substituted or unsubstituted aromatic groups having 6 to 20 carbon atoms, R, R<sup>1</sup>, R<sup>2</sup>, R<sup>3</sup>, and R<sub>A</sub> are substituted or unsubstituted aromatic groups having 6 to 20 carbon atoms or substituted or unsubstituted aliphatic groups having 1 to 20 carbon atoms, and X, X<sub>A</sub>, Y, and Z are aliphatic sulfonium groups, aliphatic sulfonium groups having fluorine, a tetrafluoroborate group, or hexafluorophosphonium group.

10 [0018]

As described above, the molecule of the photoresist base material of the invention contains a chromophore active to extreme ultraviolet radiation and/or an electron beam, the photoresist base material can exhibit performance as a photoresist by itself. Therefore, it is unnecessary to add an additive. When the composition of the invention contains a photoacid generator (PAG) or the like as a chromophore for improvement of sensitivity, the reaction may unnecessarily proceed due to generation of an excess amount of acids from the PAG, migration of the generated acids to areas other than desired areas in the photoresist thin films, and the like, resulting in impaired resolution. For this reason, when it is necessary to promote the performance as a photoresist, particularly resolution of the photoresist, a basic compound is optionally added as a quenching agent in addition to additives such as a PAG and the like.

Specifically, the quenching agent is defined as an

additive to inhibit an overreaction of PAG.

[0019]

As the quenching agent, in addition to known basic compounds, other compounds having the same effect can be commonly used. These compounds can be appropriately adjusted according to the type of substrate, a desired shape, size, and the like of minute patterns.

In the invention, it is preferable to use a basic organic compound as a quenching agent from the viewpoint of solubility in the photoresist composition and dispersibility and stability in photoresist layers.

As specific examples of the basic organic compound, in addition to pyridines such as quinoline, indole, pyridine, and bipyridine, pyrimidines, pyrazines, piperidine, piperazine, pyrrolidine, 1,4-diazabicyclo[2.2.2]octane, aliphatic amines such as triethylamine, tetrabutylammonium hydroxide, and the like can be given.

[0020]

The amount of the quenching agent to be added is usually from 25 to  $1 \times 10^{-7}$  wt% of the photoresist base material or from 50 to 0.01 wt% of the amount of PAG.

[0021]

To the extent not impairing the effect of the invention, other additives may be optionally added to the photoresist composition of the invention in addition to PAG. Such additives include a base such as tetrabutylammonium hydroxide and its salt, an anti-light splitting agent, a plasticizer, a speed promoter,

a photosensitizer, a sensitizer, an acid growth functional material, an etching resistance reinforcing agent, and the like.

[0022]

5           One of these additives may be used alone, a mixture of two or more additives with the same or different functions may be used, or a mixture of precursors of these components may be used. The composition and the amounts incorporated of these components can be appropriately adjusted according to the  
10   desired shape, size, and the like of minute patterns. In general, the same composition ratio and the like as in conventional photoresist can be applied.

[0023]

As the solvent, any solvents commonly used as a solvent  
15   for photoresist compositions can be used. Specific examples are glycols such as 2-methoxyethyl ether, ethylene glycol monomethyl ether(2-methoxyethanol), propylene glycol monomethyl ether, and acetoxymethoxypropane; lactates such as ethyl lactate and methyl lactate; propionates such as methyl  
20   propionate and ethyl propionate; cellosolve esters such as methyl cellosolve acetate; aromatic hydrocarbons such as toluene and xylene; ketones such as methyl ethyl ketone, cyclohexanone, and 2-heptanone; and the like. These solvents can be appropriately selected according to solubility of the base  
25   material in the solvent, film-formability, and the like.

[0024]

The proportion of the components other than the solvent

in the composition is appropriately determined so that a photoresist layer with a desired thickness can be formed. Specifically, the proportion is generally from 1 to 40 wt% of the total amount of the composition. This proportion, however, 5 can be appropriately adjusted according to the types of the base material and solvent, the thickness of the photoresist layer, and the like.

[0025]

The composition of the invention is uniformly applied to 10 a substrate such as a silicon wafer or any optional layers to be processed formed on a silicon wafer by spin coating, dip coating, painting, and the like. In a common practice after the application, the coated material is dried with heating at 80 to 160°C, for example, until the photoresist coating layer 15 becomes non-sticky in order to remove the solvent. The heating conditions, however, can be appropriately adjusted according to the types of the base material and solvent, the thickness of the photoresist layer, and the like.

[0026]

20 Next, the substrate on which the photoresist coating layer is no more sticky is exposed to extreme ultraviolet radiation or is irradiated with an electron beam by any optional method through a photomask to cause protective groups contained in the base material to dissociate, thereby producing solubility 25 differences between the exposed areas and unexposed areas on the photoresist coating layer. After the exposure, the substrate is baked to increase the solubility differences,

followed by development with an alkaline developer in order to form relief images. Patterns processed by ultra-microfabrication can be formed on the substrate in this manner.

5 [0027]

If the photoresist base material and the composition thereof of the invention are used, patterns with isolated lines of 100 nm or less, particularly 50 nm or less, a 1:1 line-and-space, holes, etc, can be formed at a high sensitivity, high contrast, and low line edge roughness by ultra-microfabrication using extreme ultraviolet radiation or an electron beam.

[Examples]

[0028]

15 The invention is described more specifically by way of examples. However, the following examples should not be construed as limiting the invention.

Example 1

[Photoresist base material]

20 (1) Synthesis of calix-[4]-resorcinarene

A three-neck flask (volume: 500 ml) equipped with a dripping funnel, a Dimroth condenser, and a thermometer, sufficiently dried and replaced with nitrogen gas, was encapsulated with resorcinol (33 g, 300 mmol) and acetaldehyde (17 ml, 300 mmol) in a nitrogen stream. Then, distilled methanol (300 ml) was added under a slight pressure of nitrogen gas to obtain a methanol solution. The methanol solution was

heated at 75°C on a oil bath while stirring. 75 ml of a concentrated hydrochloric acid solution was slowly added by dripping from the dripping funnel, followed by continued stirring with heating at 75°C for two hours. After completion  
5 of the reaction, the mixture was allowed to cool to room temperature, followed by cooling on an ice water bath. The reaction mixture was allowed to stand for one hour. White raw crystals of the target compound were produced and collected by filtration. The crude crystals were washed twice with purified  
10 water (100 ml), purified by recrystallization from a mixed solution of ethanol and water, and dried under reduced pressure to obtain calix-[4]-resorcinarene (16 g, yield: 40.2%), which is a compound of the formula (1) with hydrogen atoms for all Rs. The structure of this compound was identified by NMR, IR,  
15 elementary analysis, and the like.

[0029]

## (2) Synthesis of calix-[4]-resorcinarene compound

A two-neck flask (volume: 50 ml) equipped with a Dimroth condenser and a thermometer, sufficiently dried and replaced  
20 with nitrogen gas, was encapsulated with calix-[4]-resorcinarene (1.09 g, 2.0 mmol), a compound with hydrogen atoms for all Rs, prepared in (1) above, sodium carbonate (0.84 g, 7.9 mmol), and 15-crown-5 (0.63 g, 2.9 mmol). The flask was replaced with nitrogen gas. Next, after adding  
25 16 ml of acetone to prepare a solution, 2-methyl-2-adamantyl bromoacetate (1.52 g, 5.3 mmol) was added and the mixture was heated to reflux in a nitrogen atmosphere in an oil bath at 65°C



while stirring for 24 hours. The reaction mixture was allowed to cool to room temperature and filtered. The filtrate was slowly added to 40 ml of a 0.5 M aqueous solution of acetic acid to obtain a white precipitate. The precipitate was collected  
5 by filtration, washed with purified water, and dried under reduced pressure to obtain a calixresorcinarene compound (2.27 g, yield: 100%), which is a compound of the formula (1) with a 2-methyl-2-adamantyloxycarbonylmethyl group for 36% of Rs and a hydrogen atom for 64% of Rs. This compound was used as a  
10 photoresist base material. The calixresorcinarene compound was analyzed by  $^1\text{H}$ -NMR to determine the structure and the percentage of 2-methyl-2-adamantyloxycarbonylmethyl groups in Rs to confirm that the compound has the targeted structure. The  $^1\text{H}$ -NMR spectrum chart is shown in Figure 1.

15 [Industrial Applicability]  
[0030]

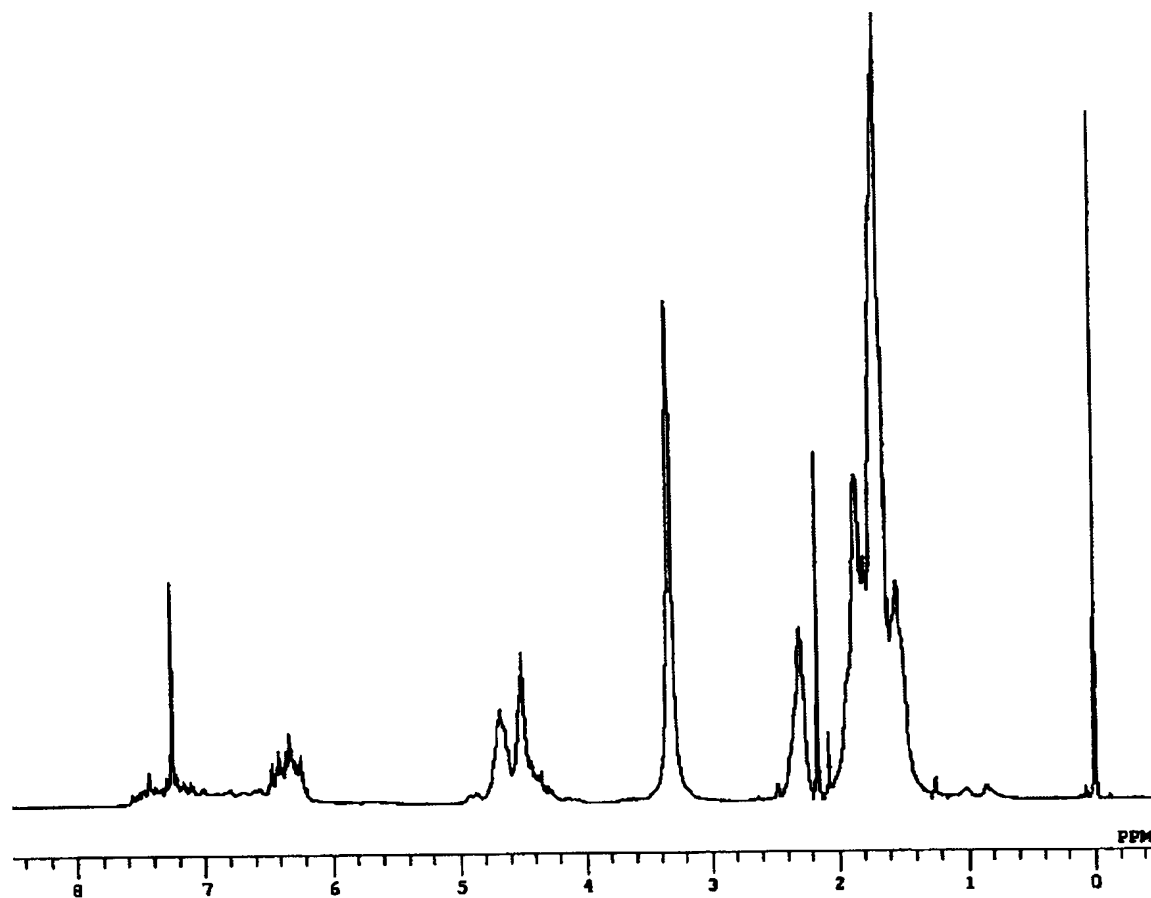
The photoresist base material and the composition thereof the invention is suitably used in the electricity and the electronic field such as a semiconductor device, the optical  
20 field, and the like. Performance of semiconductor devices such as ULSI can be outstandingly promoted by using the photoresist base material and the composition.

[Brief Description of the Drawings]  
[0031]

25 Figure 1 shows a  $^1\text{H}$ -NMR spectrum chart of calixresorcinarene compound synthesized in Example 1.

[NAME OF DOCUMENT] Drawings

[Fig. 1]

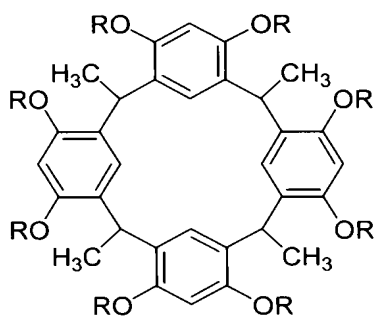


[Name of Document] ABSTRACT

[Subject] There is provided a novel calixresorcinarene compound, a photoresist base material comprising the compound enabling ultra microfabrication using extreme ultraviolet  
 5 radiation and/or an electron beam, while exhibiting high sensitivity, high resolution, and low line edge roughness, and a composition of the same.

[Solving Means] A calixresorcinarene compound shown by the following formula (1),

10



(1)

wherein R individually represents a hydrogen atom, a  
 1-tetrahydropyranyl group, a 1-tetrahydrofuryl group, or one  
 15 or more organic groups selected from the group consisting of  
 the organic groups shown by the following formulas,



hydrogen atom, a 1-tetrahydropyranyl group, and a 1-tetrahydrofuranyl group is excluded.

[Selected Drawing] FIG. 1